2D automated SEM and 3D X-ray Computed Tomography study on inclusion analysis of steels

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Abstract

In this research work, inclusion analysis was done on titanium micro-alloyed low carbon steels using automated Scanning Electron Microscopy coupled with Energy-Dispersive X-ray Spectroscopy (SEM/EDS), and 3D X-ray Computed Tomography (XCT) techniques. Automated SEM/EDS analysis provided the information about the average size, number, shape, and the composition of the inclusions. The inclusions are categorized as calcium aluminates, titanium nitrides, and “other” inclusions with an average size of 1.66 µm, 1.52 µm, and 1.31 µm respectively. To estimate the actual amount of inclusions present and to understand their 3D morphologies, XCT analysis was done at two different resolutions of 1.8 µm and 590 nm. The employment of these resolutions enabled the technique to detect huge number of inclusions with a wide size range from 0.75 µm to 201.4 µm. Moreover, the XCT technique revealed the presence of complex irregular shaped inclusions. Effective combination of these two techniques for inclusion analysis gives complete quantified information about the inclusions present in steels.

Keywords: Clean steels, Inclusion analysis, Automated SEM/EDS, X-ray computed Tomography (XCT).

1. Introduction

The occurrence of Non-Metallic Inclusions (NMI's) in steels not only pose severe casting problems [1], but also significantly deteriorate the mechanical properties of the semi-finished and the finished products [2]. In order to effectively control this NMI content, it is critical to accurately identify them through appropriate characterizing techniques which would able to generate important information such as their maximum size, size distribution, morphology, shape and chemical compositions [3]. Depending upon the time required for analysis, and the size and the type of inclusions present [4], various techniques such as optical microscopy [5], ultrasonic testing [6], laser induced spectrometry [7], optical emission spectroscopy [8], total oxygen content [9], confocal microscopy [10], and scanning electron microscopy (SEM) [7,11] are presently employed for inclusion analysis.

Among these, the most commonly used industrial method, the total oxygen content method [12], is limited only to oxide inclusions with no information about their respective shape and size distribution [9]. Another common technique is the ultra-sonic method, which can only detect inclusions of very large size of at least 20 µm [11]. With the increase in demand for ultra-clean steels and the consequential decrease in their acceptable sizes and the number densities, the above techniques can no longer be reliable in steels with multiple inclusion types. Therefore, better sensitive techniques which can give complete information about the inclusions present in the cleaner steels are needed to be employed. However, it should also be noted that no single technique can effectively give this complete information [11]. Therefore, in this research work, we propose the use of automated Scanning Electron Microscopy analysis coupled with Energy-Dispersive X-ray Spectroscopy (SEM/EDS) [13] and three dimensional (3D) X-ray Computed Tomography (XCT) techniques for the inclusion analysis of clean steels.

2. Experimental Procedure

2.1. Material

The present work was conducted on calcium treated Titanium micro-alloyed low carbon steel. The chemical composition of the lollipop sample taken after the ladle treatment was found to be Fe-0.06C-0.0068N-1.96Mn-0.0016S-0.1Si-0.04Al-0.13Ti-0.0034Ca (wt %).

2.2. Automated SEM/EDS analysis

A sample was cut from the lollipop sample, and mounted and polished using standard metallographic procedures. Automated SEM/EDS analysis was done on an area of 20.3 mm^2 using FEG JEOL 7800F-SEM. For simultaneous EDS analysis, an upper limit on the amount of Fe and C content and a lower limit on the total amount of components that can be present in inclusions was set in Aztec Software (Oxford instruments, UK).

2.3. 3D XCT analysis

Two samples of dimensions 0.5X0.5X1.5 mm^3 and 1.5X1.5X3 mm^3 were cut using wire EDM cutting machine to obtain voxel resolutions of 519 nm and 1.8 µm respectively. These samples were then individually scanned using Zeiss Xradia 520 Versa XCT machine. The XCT scanning settings are shown in the **Table I**.

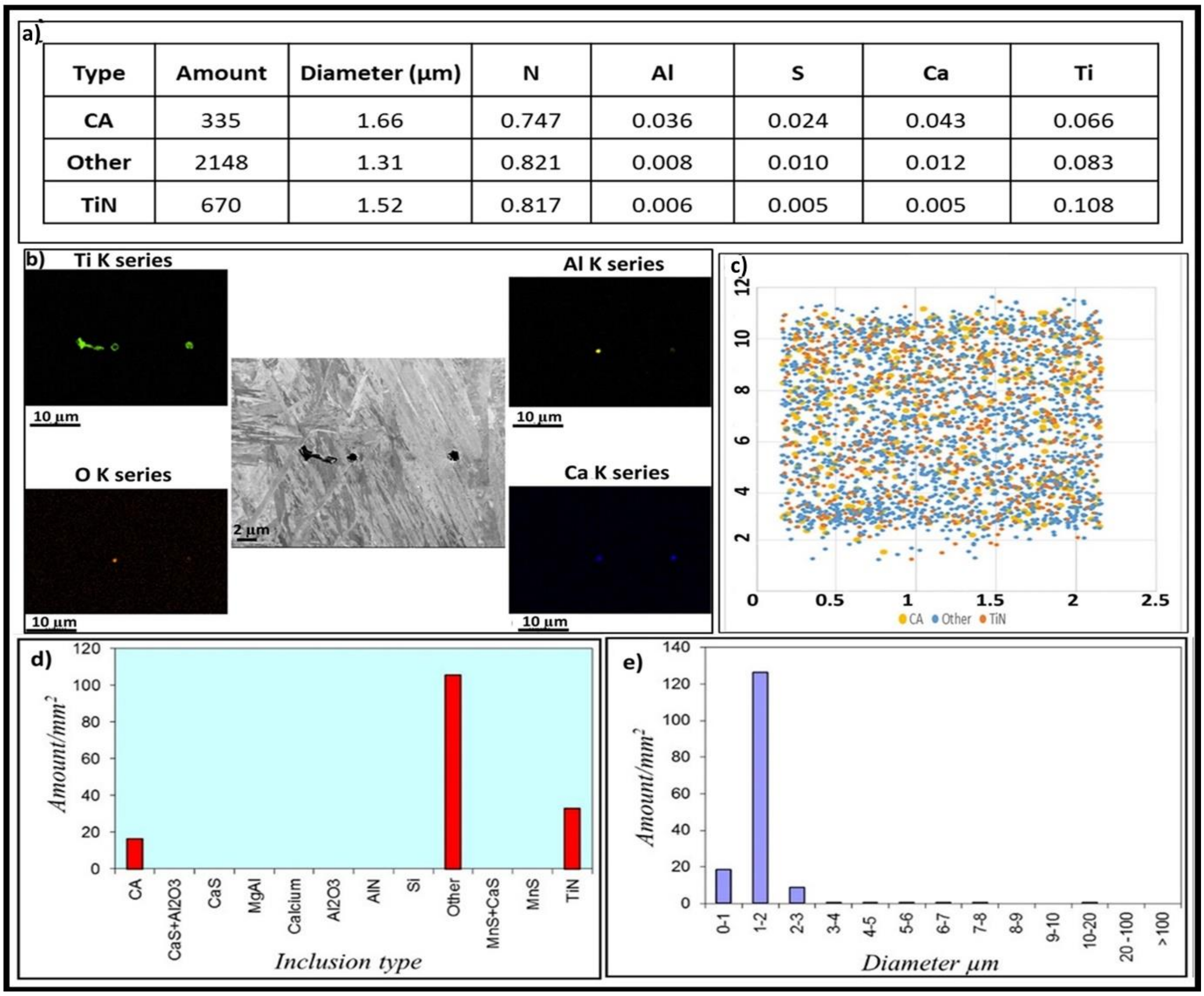
Table I: XCT parameters used in this study.

Equipment Used: Zeiss Xradia 520 Versa		
Parameters		
Sample dimension (mm^3)	*0.5 X 0.5 X 1.5*	*1.5 X 1.5 X 3*
Exposure Voltage (kV)	155	155
Exposure Power (W)	9.9	9.9
Exposure Time (sec)	17	10
Optical Magnification	10x	4x
Information about the scan		
Voxel size (µm)	0.595	1.86
No of Projections	3201	3201

3. Results and Discussion

3.1. Automated SEM/EDS analysis

Figure 1 shows the automated SEM/EDS analysis of the steel sample. Three types of inclusions were detected: 1. Calcium aluminates (CA), 2. Titanium nitrides (TiN), and 3. “Other” inclusions. However, the inclusions which are categorized as “Other” inclusions can also be considered as TiN inclusions as the criteria for screening the TiN type was set to a minimum of 0.1 atomic fraction of titanium. The “Other” types have a slightly lower titanium content (~0.083 at %). The total number, the average size and the compositions of these three inclusion types are shown in **Figure 1 a)**. The average diameter of CA, TiN and others were found to be 1.66 µm, 1.52 µm, and 1.31 µm respectively. The presence of complex inclusions like CaO-Al_2O_3-TiN and Al_2O_3-TiN can be clearly seen from the **Figure 1 b)**. However, this technique completely disregarded the occurrence of these complex inclusions as it calculates the chemical composition by taking the mean of the chemical elements detected in each inclusion. The exact position of each inclusion, their respective type, and size distributions are shown in **Figure 1 c), d), and e)** respectively**.** The number density of CA, TiN and “other” inclusions were found to be 17, 33, and 106 per mm^2 respectively. The size distribution reveals that the majority of inclusions have size less than 3 µm with very few inclusions above 5 µm. These similar size ranges for identical steel compositions were well published in the literature [14].



Type	Amount	Diameter (µm)	N	Al	S	Ca	Ti
CA	335	1.66	0.747	0.036	0.024	0.043	0.066
Other	2148	1.31	0.821	0.008	0.010	0.012	0.083
TiN	670	1.52	0.817	0.006	0.005	0.005	0.108

Figure 1: *Automated SEM/EDS analysis: a) Total amount, average size, and composition (at fraction), b) SEM/EDS image, c) Position, d) Type distribution, and e) size distribution of inclusions.*

All the above information obtained from this technique comes from a limited space of a single 2D surface. Due to this the real shape, number and size of the inclusions can be very different from the obtained results [11,15,16]. Moreover, as the stress concentration effect of these NMI's are highly dependent on their shapes, it is absolutely necessary to understand their real 3D shapes and sizes [17].

3.2. 3D XCT analysis

Figure 2 a) shows the 3D reconstructed inclusion distribution in the steel samples measured at resolutions of 1.8 µm and 590 nm. When compared to the 2D morphologies (**Figure 1 b**), the 3D morphologies of the inclusions clearly show the complexity of their irregular shapes. This demonstrates that the shape and number of inclusions obtained from 2D SEM analysis can be very different from the actual volumes of the samples. For instance, an irregularly shaped inclusion shown through the arrows in **Figure 2 a)** can be easily misinterpreted as a cluster of inclusions on the 2D surface. This can hugely distort the inclusion analysis obtained from the 2D SEM analysis. Moreover, the shapes of these irregular inclusions viewed in SEM analysis can vary dramatically with the change in the position and direction of the viewing plane. **Figure 2 b)** and **c)** shows the size distribution of inclusions in steel samples measured at different resolutions of 1.8 µm and 590 nm respectively. Large number of macro-inclusions with sizes greater than 5 µm were found in this analysis, whereas very few large inclusions were detected in automated SEM analysis. The sizes of the inclusions ranged from 0.75 µm to 201.4 µm. This size range is broad when compared with the size range of 27-45 µm obtained by Christian et al., for their inclusion analysis of quench and tempered steels (different composition) using XCT technique [18]. The largest inclusion size detected by this technique was 201.4 µm, whereas the largest inclusion size detected by automated SEM analysis was 19.6 µm. This is because the XCT technique captures the inclusions from the whole volume, whereas the automated SEM/EDX analysis is a surface technique. Moreover, depending upon the magnification used in the automated SEM analysis, the large inclusion present on the 2D surface can be easily considered as more than one inclusion if it falls on more than one plane during the scanning process. Additionally, there is no chance of erosion in the XCT analysis as the metallographic polishing of samples is not required here. However, at lower resolutions, XCT technique can misinterpret a cluster of closely packed inclusions as a single large inclusion. From **Figure 2** it is also evident that a huge number of inclusions are detected in the sample volume. This will allow the statistical methods to accurately predict the actual scenario in the steels [17]. Therefore, the use of this technique for inclusion analysis will allow the steel manufacturers to understand the true nature of the inclusions present in steels. However, unlike automated SEM/EDS analysis, XCT analysis gives no information regarding the chemical compositions of the inclusions.

Therefore, the effective combination of 2D automated SEM/EDS and 3D XCT techniques provides information such as number density, size distribution, maximum size, 3D shape and morphologies, position and chemical compositions of the inclusions. Extensive use of these techniques can lead to a better understanding of the inclusions present in steels and hence assist in developing beneficial microstructures [19] with enhanced mechanical properties [20].

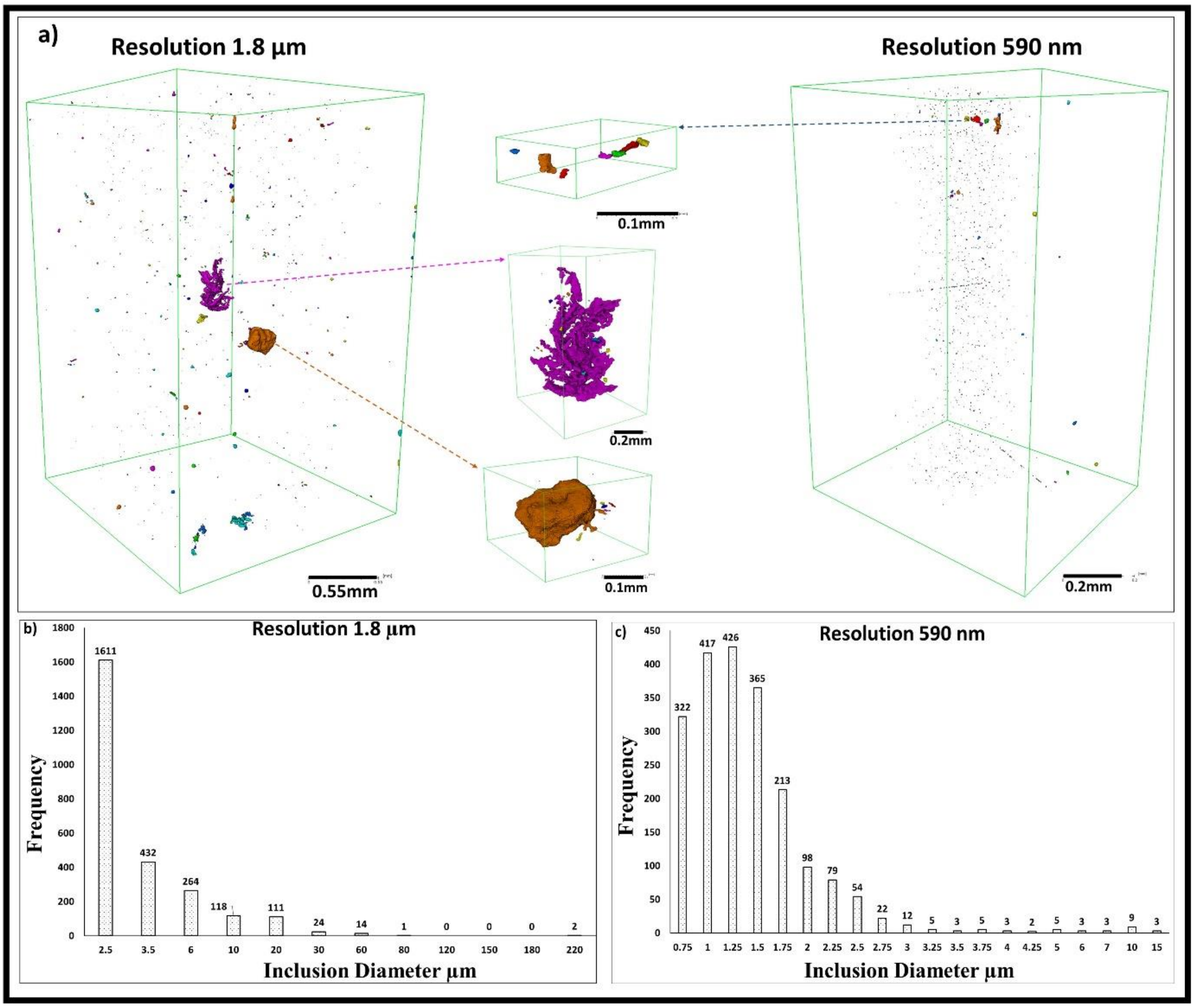


Figure 2: *XCT analysis: a) 3D reconstructed inclusions and size distribution of inclusions measured at b) 1.8 µm and c) 590 nm resolutions.*

4. Summary

2D automated SEM and 3D XCT techniques were used to quantify the inclusions present in calcium treated titanium micro-alloyed low carbon steel. 2D automated SEM/EDS analysis showed that the type of inclusions were found to be calcium aluminates, titanium nitrides and "other" inclusions with an average size of 1.66 µm, 1.52 µm, and 1.31 µm respectively. 3D XCT analysis provided the number densities, average size and 3D morphologies of the inclusions. When compared to automated SEM/EDS, XCT technique detected a huge number of inclusions with their corresponding 3D shapes. A wider size range of inclusions (0.75 µm 201.4 µm) were detected through this technique. However, there is no information obtained regarding the composition of the inclusions. Effective combination of these two techniques can be useful in predicting the actual scenario in the steels.

5. References

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